

SUGARCANE FACTORY PERFORMANCE OF COLD, INTERMEDIATE, AND HOT LIME CLARIFICATION PROCESSES

GILLIAN EGGLESTON^{1,3}, ADRIAN MONGE² and BLAINE E. OGIER¹

¹USDA-ARS-Southern Regional Research Center
1100 Robert E. Lee Blvd.
New Orleans, LA 70124

²Cora Texas Manufacturing Co.
Res. 32540 B Texas Rd.
White Castle, LA 70788

Accepted for Publication November 1, 2002

ABSTRACT

A comparative factory investigation of hot versus intermediate and cold lime clarification was undertaken to quantify performance. In cold liming, mixed cane juice (MJ) was incubated (8 min) and then limed in a lime tank (4 min), both at ~105F. For intermediate liming, 50% of the MJ was heated (180-200F) before incubation (8 min), then limed in a lime tank (4 min) at ~150F. Hot liming was configured very similar to intermediate liming except that incubation time was increased from 8 to 12 min, and that lime was added immediately after flash-heating (215F; 30 s). Overall, both hot and intermediate liming performed much better than cold liming, and hot liming offered some extra advantages over intermediate liming. Less sucrose was lost to inversion reactions across both hot (season avg. 0.79%) and intermediate (0.97%) lime processes than across cold liming (1.48%). By operating hot liming, the reduction in sucrose losses alone saved the factory approximately US \$283,000 over cold liming. Increasing the factory target pH of the final evaporator syrup from ~pH 6.0 to 6.3, in both hot and intermediate liming, markedly reduced sucrose inversion losses across the clarifiers and evaporators. Dramatically less lime had to be added in hot liming compared to either cold or intermediate liming, with the factory consuming, on season average, only 1.01 lbs lime/ton cane compared to 1.28 for the previous grinding season when intermediate rather than hot liming was operated. Preheating 50% of the MJ in both intermediate and hot liming consistently removed color, dextran, and starch, but silicate levels were not significantly

³ Author to whom all correspondence should be sent: Dr. Gillian Eggleston, USDA-ARS-Southern Regional Research Center, 1100 Robert E. Lee Blvd., New Orleans, LA 70124. TEL: 504-286-4446; FAX: 504-286-4367; E-mail: gillian@srcc.ars.usda.gov

changed. Although the fastest settling occurred in intermediate liming, ~2.1% (season avg.) more turbidity removal (MJ to clarified juice [CJ]) occurred in both hot and intermediate liming compared to cold liming. Markedly less color formed and dextran removal was the best across hot liming. Using hot liming across the season, the factory observed 12-15% more heating capacity in the limed juice heat exchangers and a 90% reduction in the quantity of chemicals needed to clean the heat exchangers.

INTRODUCTION

The degree of clarification has a great impact on boiling house operations, sugar yield, and refining quality of raw sugar. Several lime-clarification systems have been developed over the years including cold, hot, intermediate, fractional, and sacchararate liming. Moreover, variations also occur within a particular clarification system, from factory to factory. Although many other parts of the world have changed from cold liming, mostly to hot liming, cold liming is still usually operated in the U.S. The main advantages of cold liming over other liming processes have been considered to be its simplicity of operation and less sucrose inversion (Chen 1993), but these conclusions were drawn mainly from laboratory studies, which do not always reflect the complexity of factory processing streams which can change in seconds, and give little or no information on process control which is essential for engineers. Recent factory studies (Eggleston *et al.* 1999, 2002; Eggleston 2000a, b) have unequivocally shown that excessive inversion occurs in cold liming clarifiers, excessive color is formed on liming, pH and turbidity control are erratic, and turbidity removal is not adequate. Furthermore, with the introduction of mechanical harvesting of green and burnt billeted sugar cane in the 1990s in the U.S., especially in Louisiana, there has been an unfortunate large increase of impurities that require factory processing. Therefore, there is currently an even greater need to remove these extra impurities during clarification by using more advanced clarification systems than cold liming.

Although Eggleston (2000a, b) previously compared the performances of hot and cold lime clarification systems at two Louisiana factories across the 1998 grinding season, and compared intermediate and cold lime processes at a third Louisiana factory across the 1999 season (Eggleston *et al.* 2002), a systematic factory comparison of all three processes has not been reported. Such a study would be useful to help processors decide which clarification process is best to utilize; consequently, this study was undertaken to compare the performance of cold, intermediate, and hot lime clarification processes in a factory processing mostly billeted cane. As numerous potential benefits of intermediate liming over cold liming had been observed in a previous grinding season study at the factory

in this study just by preheating only 30% of the mixed juice before incubation and lime addition, for this study the factory increased the amount preheated to 50%. Also, unlike previous factory studies, in this study raw sugar samples were analyzed to assess the impact of the clarification process on the raw sugar quality. The factory in this study also operated an incubator tank, for the application of dextranase when the factory was suffering from dextran problems, and to allow the natural diastase enzyme in the juice to degrade starch (Eggleston *et al.* 2002).

MATERIALS AND METHODS

This study was performed at Cora Texas raw sugar factory, Louisiana, across the 2000 grinding season. In an attempt to further improve clarification performance, the factory made the decision to convert to hot lime clarification during the 2000 grinding season. The season average cane grinding rate and flowput were 521 short tons/h and ~1900 gallons/min, respectively, and ~99% of the cane processed was billeted, of which ~75% were green billets. All mixed juice was prescreened.

Factory Clarification Equipment and Procedures

The flow diagram for hot lime clarification is illustrated in Fig. 1a. To convert to hot lime clarification the factory had to install new equipment, including a lime injector 4 in. below the bottom of the flash tank (Fig. 2), a static mixer, and new pH measurement instrumentation to measure the pH of the flash heated limed juices at high temperatures. Factory measurement of pH was done by taking a continuous sample of the flash-heated limed juice and passing it through a cooling column before the pH measurement was made with a Van LondonTM industrial electrode. In hot liming (Fig. 1a), 50% of the MJ was pumped to heaters and heated to 190-200F before entering a juice incubation tank operated at ambient temperature (retention time 12 min). The remaining 50% of "nonpreheated or cold" MJ was pumped directly into the incubator tank. Filtrate from the clarifier mud filters, was also added in the incubation tank. This mud filtrate is produced when the precipitated mud in the clarifiers is filtered through rotatory vacuum filters and is then recirculated into the incubation tank to recover as much sucrose as possible. The incubated juice was then flash heated to ~218-220F to maintain constant temperature and remove air bubbles. Lime (~12 Baume) was injected automatically into the flash heated juice 4 in. below the flash heater, mixed and then distributed into one of four clarifiers (Fig. 2). Polyelectrolyte flocculants were added (4 ppm on clarified juice) before entering the clarifiers. In this study clarified juice was taken from

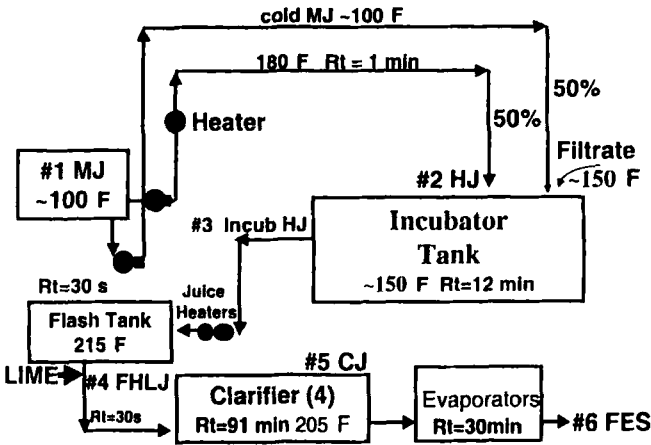


FIG. 1A. FLOW DIAGRAM OF THE HOT LIME CLARIFICATION PROCESS
MJ=mixed juice; HJ=heated juice; Incub HJ=incubated heated juice; FHLJ=flocculated heated limed juice; CJ=clarified or clear juice; FES=final evaporator syrup out of the last evaporator body.

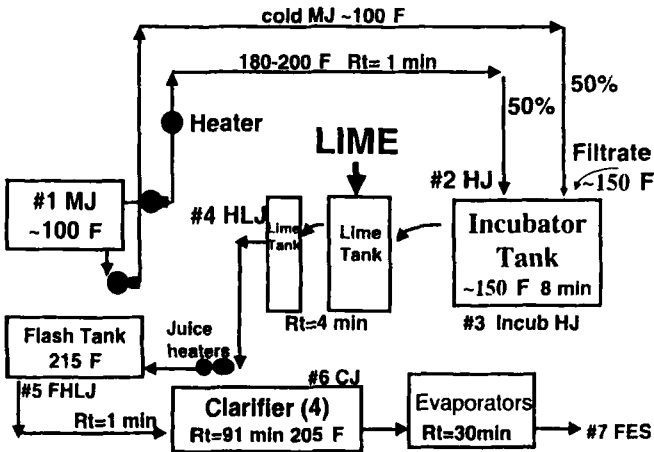


FIG. 1B. FLOW DIAGRAM OF THE INTERMEDIATE LIME CLARIFICATION PROCESS
MJ=mixed juice; HJ=heated juice; Incub HJ=incubated heated juice; HLJ=heated limed juice; FHLJ=flocculated heated limed juice; CJ=clarified juice; FES=final evaporator syrup.

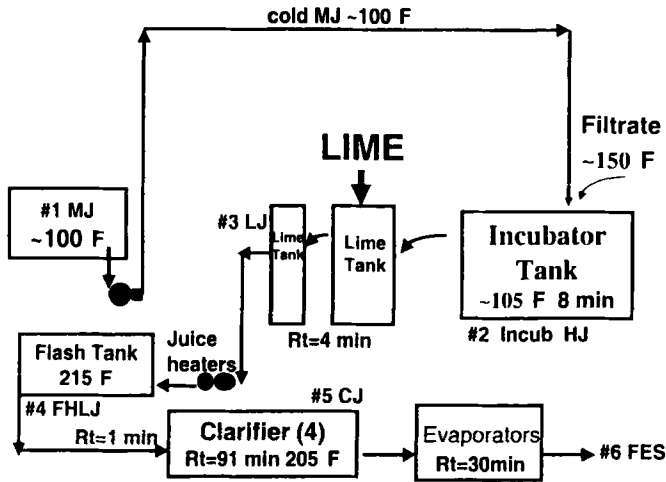


FIG. 1C. FLOW DIAGRAM OF THE COLD LIME CLARIFICATION PROCESS
MJ=mixed juice; Incub HJ=incubated heated juice; LJ=limed juice; FHLJ=flocculated heated limed juice; CJ=clarified juice; FES=final evaporator syrup.

the #4 Dorr Oliver 444 clarifier. The evaporation station consisted of two preevaporators and three triple-effect Robert's type Calandria evaporators. Commercial α -amylase (2.5 lbs/500 tons of cane) was added in the last bodies of the triple effect evaporators (max. temp 150F).

In case of difficulties in hot lime clarification or cane quality problems, the factory still wanted the ability to revert to intermediate and cold liming when necessary, which was convenient for this study. Flow diagrams of the factory intermediate and cold lime clarification processes are shown in Fig. 1b and c, respectively. For the conversion to intermediate liming from hot liming, two of the three tanks comprising the collective incubation tank were used as lime tanks ($R_t = 4$ min) and pH measurement occurred there.

Cold liming was the same as intermediate liming, except that the recirculation pump after the mixed juice tank was stopped so that all the MJ entered the incubation tank without first being preheated (Fig. 1c). For all three clarification processes, the target pH of the final evaporator syrup was usually 6.0.

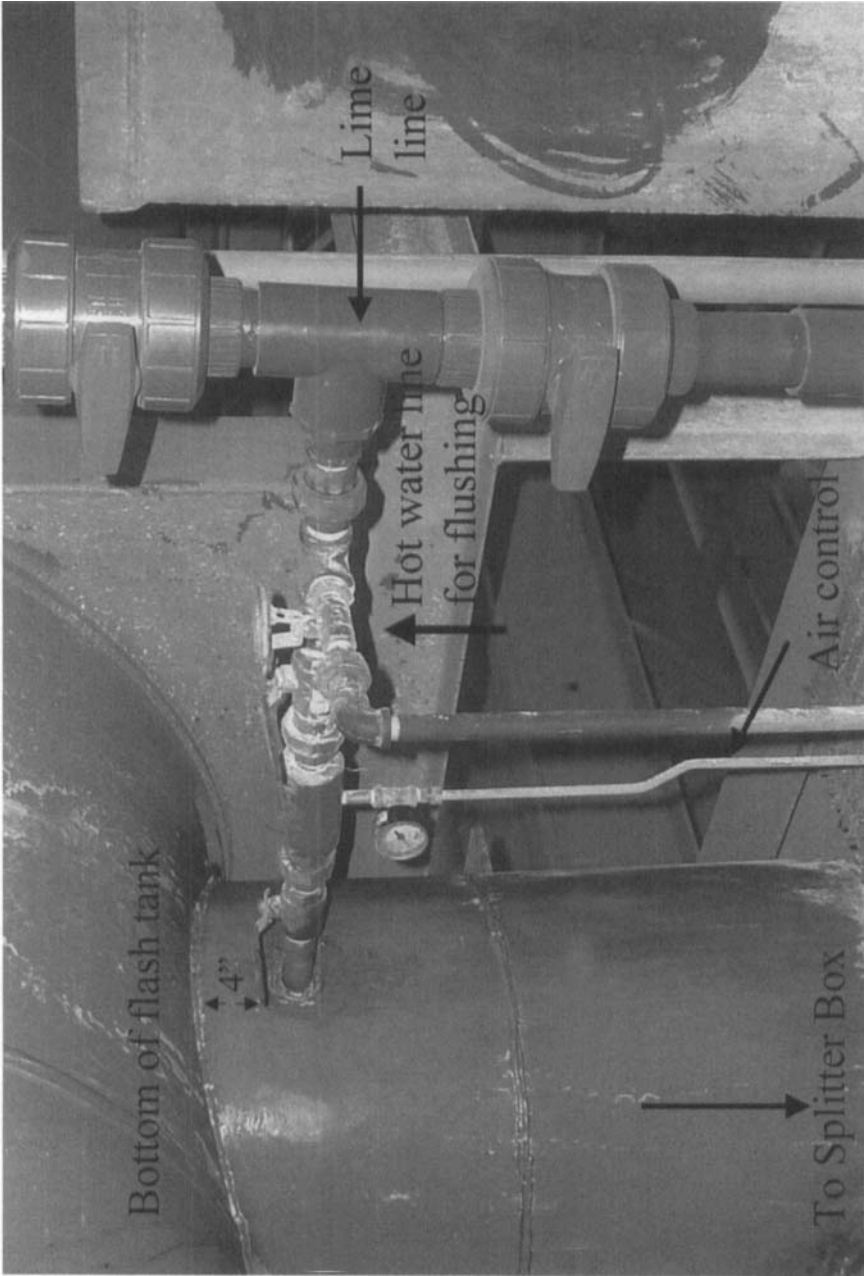


FIG. 2. DIAGRAM OF THE LIME INJECTION EQUIPMENT FOR HOT LIME CLARIFICATION

General Sampling

Because stored cane at the factory deteriorates more rapidly overnight, samples across cold, intermediate, and hot liming were taken between 8 am - 5 pm, on three consecutive days, respectively. The factory converted to cold or intermediate liming at least 1 h prior to sampling to flush out the hot lime juice streams. Juices and syrups were carefully collected to prevent further chemical degradation reactions and/or microbial growth. Each sample was first collected in a large (250 mL) container, and then ~25 mL was poured into a 50 mL container. Sodium azide (0.02%) was added to the 50 mL container before putting in dry ice. Glucose, fructose, and sucrose concentrations were measured in juice from the small containers, usually the next day. Juice in the large containers was immediately cooled on ice, and Brix and pH were then measured at the factory. Sodium azide was then added and the juice stored on dry ice until transportation to, and storage in, a -80C (-112F) laboratory freezer, subsequent to laboratory analyses. Flow rates in any factory fluctuate constantly, therefore, hourly samples were taken across a sampling period and grinding season to obtain precise averages. A six hour sampling period across each clarification system was repeated three times across the grinding season, in order to cover cane variety, environmental, and process parameter variations. The three clarification sampling period dates were: sampling period one, cold 3 Oct and intermediate 4 Oct; sampling period two: cold 8 Nov, intermediate 9 Nov and hot 10 Nov; sampling period three: cold 12 Dec, intermediate 13 Dec and hot 14 Dec.

Hot Lime Sampling

Mixed juice (MJ), heated juice (HJ), incubated juice (incub J), flocculated heated limed juice (FHLJ), clarified juice (CJ), and final evaporator syrup (FES) were collected hourly over a six hour period (Fig. 1a). Retention times in the pipes and tanks were taken into account. Consequently, there was a 1 min delay between sampling MJ and HJ, a 12 min delay between HJ and incub J, a 30 s delay between incub J and FHLJ, a 91 min delay between sampling FHLJ and CJ (residence time [Rt] in the clarifier was calculated using tank dimensions and average flow rate), and a further 30 min delay between sampling CJ and FES, which was only an approximation. Because the factory had mechanical problems with the new hot liming pH system at the beginning of the grinding season, no valid hot liming samples were taken for the first sampling period.

Intermediate Lime Sampling

Sample collection was the same as for hot liming except that the three incubator tanks in hot liming were converted to one incubator tank ($R_t = 8$ min)

followed by two lime tanks (combined $R_t = 4$ min), which is illustrated in Fig. 1b. Consequently, a sample of heated limed juice (HLJ) was taken after incubation (Fig. 1b).

Cold Lime Sampling

Sample collection was the same as for intermediate liming, except there was no heated juice (HJ) sample as cold mixed juice was pumped directly into the incubator tank (Fig. 2c).

Raw Sugar Sampling

Raw sugar samples from each clarification process were also collected, at random, ~3 h after sampling began, and stored in a desiccator before analyses.

Sucrose, Glucose and Fructose Concentrations

The determination of sucrose, fructose and glucose in cane juices and syrups by GC was based on ICUMSA method GS7/4-22 (1998) with modifications by Eggleston *et al.* (2002).

Calculation of Sucrose Losses

Percent of sucrose losses were calculated using the following formula of Schaffler *et al.* (1985):

$$\% \text{ Sucrose lost} = \frac{\left(\frac{(\% \text{ Glc})_{\text{out}}}{\text{Brix}} - \frac{(\% \text{ Glc})_{\text{in}}}{\text{Brix}} \right) \cdot \text{MW}_{\text{SUC}} \times 100}{\frac{(\% \text{ Suc})_{\text{in}}}{\text{Brix}} \times \text{MW}_{\text{Glc}}}$$

where: MW = molecular weight, Suc = sucrose, Glc = glucose

Settling Rates and Mud Volumes of Flocculated Heated Limed Juices

See Eggleston *et al.* (2002) for general settling and mud volume measurements and calculations. The flash-heated limed juice with flocculant added was brought to a boil before settling measurements were undertaken, in order to remove interfering gas bubbles.

Brix, pH, Color, and Turbidity

Mean Brix of triplicate samples was measured using an Index Instrument temperature controlled Refractometer accurate to ± 0.01 Brix. The pH was measured at room temperature ($\sim 25^\circ\text{C}$ or 77°F), using an Ingold™ combination pH electrode connected to a Metrohm 716 DMS pH meter. Color and turbidity were measured at 420 nm and calculated according to the official ICUMSA method GS2/3-9 (1994). Samples (5 g) were diluted in triethanolamine/hydrochloric acid buffer (pH 7) and filtered through a 0.45 μm filter.

Dextran and Starch

Dextran and starch concentrations were determined for duplicate composite samples (10 g of each hourly samples were combined). Dextran was measured using the ASI-II (Sarkar and Day 1986) method, and starch was measured using a colorimetric method (Godshall *et al.* 1991), based on the starch-iodine complex.

Calcium and Silicate

Calcium and silicate concentrations in composite samples were measured. Calcium as CaO was measured by EDTA titration following ICUMSA method GS8/2/3/4-9 (1994), and silicate as Si was measured by atomic absorption spectrometry.

Analysis of Data

Data were analyzed using PC-SAS 8.1 (SAS Institute, NC) software. Process (intermediate, cold and hot liming) and sample type were considered as fixed effects. Means comparisons were undertaken using Duncan's New Multiple Range Test.

RESULTS AND DISCUSSION

Effect of the Three Different Lime Clarification Systems on Color Removal and Formation

For all three clarification systems, the color of the incoming mixed juice (MJ) differed little at the beginning and middle of the season, but decreased slightly at the end of the season. As illustrated in Fig. 3, for hot and intermediate liming, preheating 50% of the mixed juice (HJ) before incubation caused marked color (season avg. $\sim 26\%$) removal, which agrees with previous studies (Muller 1921; Eggleston 2000a and Eggleston *et al.* 2002). Furthermore, color

removal in the HJ caused the incub J color to be lower than in cold liming (Fig. 3). This heat induced color removal is considered to be associated with the precipitation of macromolecules, including polysaccharides (starch and dextran) and proteins (Armas *et al.* 1999). Color was also generally removed on incubation (Fig. 3) in all three processes, which is most likely because of precipitation with lime salts in the added filtrate juice.

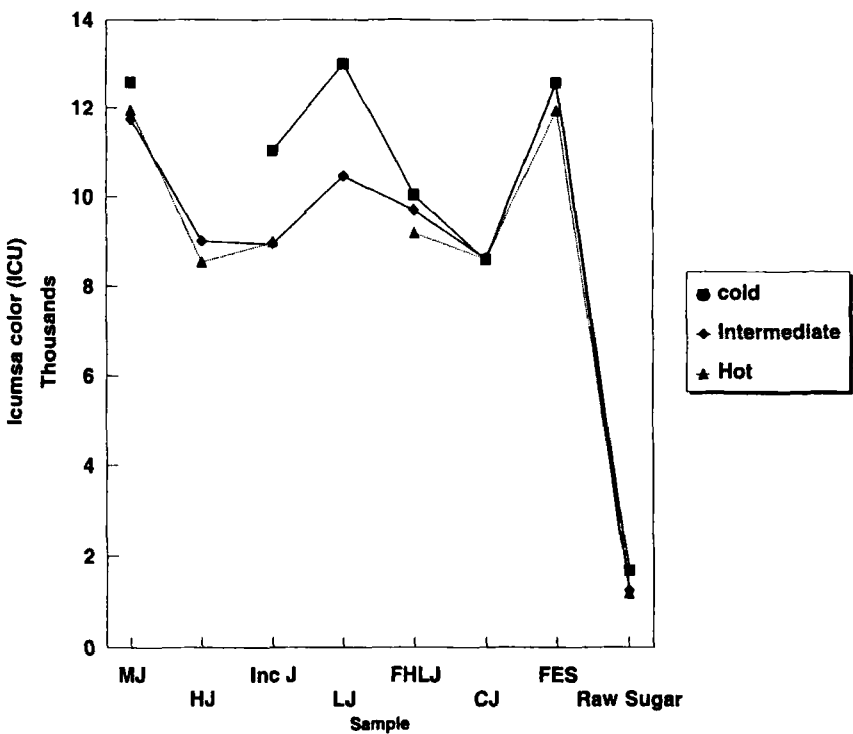


FIG. 3. AVERAGE COLOR REMOVAL AND FORMATION AMONG THE THREE CLARIFICATION PROCESSES

MJ=mixed juice; HJ=heated juice; Incub J=incubated juice; LJ=limed juice; FHLJ=flocculated heated limed juice; CJ=clarified juice; FES=final evaporator syrup.

Color is formed on liming because of the alkaline degradation of invert, a reaction that is relatively fast, and increases with temperature and retention time Rt. Because there was a 4 min Rt of liming in tanks for both cold and intermediate liming, ~16% color formation occurred. In hot liming, lime was

added directly into the juice pipe just after flash heating (Fig. 2) and mixed with static mixers. This had the effect of reducing color formation to a range of only 1.7-2.8% across the season. However, in a previous study of hot liming at another Louisiana factory by Eggleston (2000a), no measurable color was formed.

As expected, for all three processes color was removed by the settling process in the clarified juice (CJ) samples, and was formed across the evaporators because of the further reactions of sucrose inversion products. Although there were very little differences in CJ color for all three processes, final evaporation syrup (FES) and raw sugar colors were markedly lower in hot liming, and color control was better too (Fig. 3).

Lime Consumption

Lime consumption was dramatically less in hot liming than both cold and intermediate liming, and is one of the major advantages of operating hot lime clarification. Relative lime addition was measured indirectly as the difference between the calcium concentrations in the mixed juice samples and in the samples where lime was added in the factory (Table 1). Much less lime had to be added in hot liming compared to either cold or intermediate liming. This dramatic difference was also reflected in the factory season average data. With the new operation of hot lime clarification across the 2000 grinding season, the factory consumed only 1.01 lb lime/ton cane compared to 1.28 lb lime/ton cane it consumed for the 1999 grinding season when intermediate liming was operated. Furthermore, even across the next grinding season in 2001 when hot liming was still operated but the factory target FES pH was increased to pH 6.3 from 6.0, still only 1.05 lb lime/ton cane was consumed. It must also be noted that in sampling period 1, for both cold and intermediate liming (no data was available for hot liming), lime addition was much higher (Table 1). This is not really surprising because at the beginning of the grinding season, the pH of the extracted juice was unusually low (sometimes less than pH 4.5) which caused the factory to add caustic soda and lime in the cane wash water, but also necessitated the addition of more lime at the clarification stage.

Effect on Silicate Levels

Previous research (Muller 1921) on hot liming systems conducted in the laboratory suggested that preheating of the juice before adding lime caused the precipitation of silicate. This would have an enormous impact in the factory as silicate contributes largely to the scaling in evaporators. Consequently, we decided to measure silicate levels in composite samples taken across the different clarification systems and results are listed in Table 2. Juice was preheated before liming only in the hot and intermediate clarification systems, however, silicate

levels in the heated juice sometimes decreased as well as increased. Furthermore, there were no significant differences among silicate values in the final evaporator syrups for any of the three clarification systems. However, this may be because not all the mixed juice was preheated before intermediate and hot liming and measurements in three composite samples across the grinding season may not properly reflect real factory effects.

TABLE 1.
CALCIUM CONCENTRATIONS IN COMPOSITE SAMPLES

Clarification Process / Sampling Period	Ca as CaO (ppm/Brix basis)		
	MJ	LJ/HLJ/FHLJ	Change MJ to LJ/HLJ/FHLJ*
<u>COLD</u>			
1	21.47	53.37	31.9
2	21.64	37.09	15.45
3	22.57	38.76	16.19
Average:			21.18
<u>INTERM.</u>			
1	24.22	52.10	27.88
2	19.37	39.62	20.25
3	22.24	39.68	17.44
Average:			21.86
<u>HOT</u>			
1	--	--	--
2	22.83	25.14	3.03
3	19.75	32.46	12.71
Average:			7.87

*These are the samples where lime was added in their respective clarification process

pH and Sucrose Loss Control

Changes in pH across each process and sampling period are illustrated in Fig. 4. Patterns of changes in sample pH across each process were similar to

those previously reported (Eggleston 2000a; Eggleston *et al.* 1991, 2002). In general, the pH of the CJs, FESs, and raw sugars were slightly higher in intermediate and hot liming than cold liming, which affected sucrose losses and profits (Table 3). In the third sampling period at the end of the season, the effect of a higher target pH for the final evaporator syrup (FES) was studied in order to evaluate the impact on sucrose losses and economical profits. Across the 2000 grinding season the factory target pH of the FES was 6.0 but in this study it was increased to pH 6.3 in the final sampling period for both intermediate and hot liming (the effect was not studied on cold liming because of the clear disadvantages of operating this process). As can be seen in Fig. 4, increasing the target FES pH caused pH increases in the FHLJ and subsequent samples; furthermore, sample pHs were slightly higher in hot liming than in intermediate liming even though less lime had to be added.

TABLE 2.
SILICATE CONCENTRATIONS IN COMPOSITE SAMPLES

Clarification Process / Sampling Period	Si as Silicate (ppm/Brix basis)			
	MJ	HJ	CJ	FES
<u>COLD</u>				
1	51	n/a	6.7	9.6
2	91.4	n/a	10.2	11.0
3	138	n/a	9.9	9.5
Average \pm Std. Dev	93.5 \pm 43.5		8.9 \pm 1.9	10.0 \pm 0.8
<u>INTERMEDIATE</u>				
1	48.3	59	8.7	10.0
2	310	247	7.5	13.4
3	109	127	10.4	12.1
Average \pm Std. Dev	155.8 \pm 137.0	144.3 \pm 95.2	8.9 \pm 1.5	11.8 \pm 1.7
<u>HOT</u>				
1	--	--	--	--
2	115	107	13.5	10.3
3	96	104	14.8	10.3
Average \pm Std. Dev	105.5 \pm 13.4	105.5 \pm 2.1	14.2 \pm 0.9	10.3 \pm 0.0

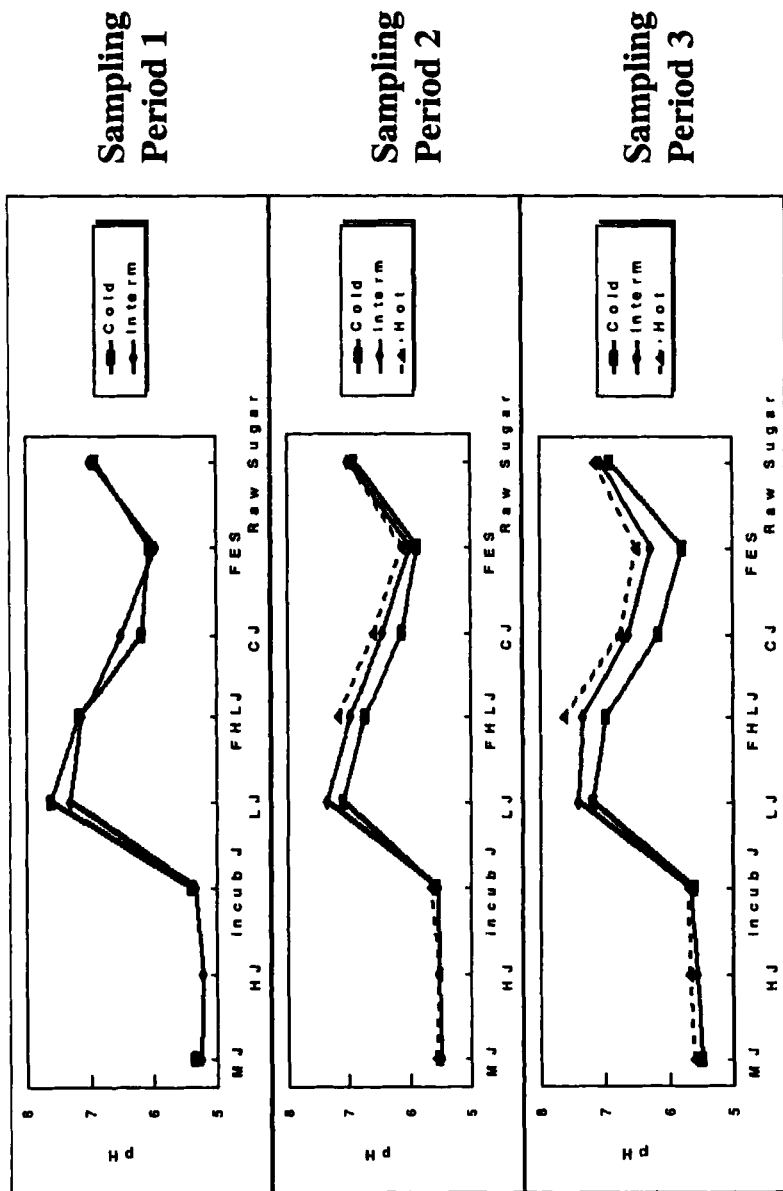


FIG. 4. VARIATIONS IN AVERAGE pH AMONG THE THREE CLARIFICATION PROCESSES

The target pH in sampling period 3 for intermediate and hot liming was increased from the usual value of pH 6.0 to 6.3. MJ = mixed juice; HJ = heated juice; Incub J = incubated juice; LJ = limed juice; FHLJ = flocculated heated limed juice; CJ = clarified juice; FES = final evaporator syrup

TABLE 3A.
% SUCROSE LOSSES ACROSS THE THREE CLARIFICATION PROCESSES

Sampling Period	Clarification Process	% Average Sucrose losses ^a				
		Across the Incubator Tank	Across the Flash Heater	Across the Clarifier Tank	Across the Evaporator Station	Total
1	Cold	0.0	0.64	0.55	1.03	2.22
	Interm	0.0	0.33	0.19	0.90	1.41
	Hot	--	--	--	--	--
2	Cold	0.0	0.25	0.71	0.23	1.19
	Interm	0.0	0.0	0.88	0.32	1.2
	Hot	0.25	n/a	0.41	0.64	1.30
3	Cold	0.0	0.40	0.42	0.25	1.07
	Interm	0.0	0.40	0.46 ^b	0.09 ^{b,c}	0.55 ^b
	Hot	0.0	n/a	0.11 ^b	0.0 ^{b,c}	0.11 ^b

^a Sucrose losses calculated according to the formula in the Materials and Methods section

^b The target pH of the final evaporator syrup was increased from 6.0 to pH 6.3

^c These figures do not take into account any glucose formed from inversion in pre- and intermediary evaporator bodies, which may have been further degraded or removed by precipitation causing reported sucrose losses across the evaporator station to be underestimates.

TABLE 3B.
SEASON AVERAGE U.S. DOLLAR LOSSES ACROSS THE THREE CLARIFICATION PROCESSES

	Clarification Process	Lost U.S. Dollar Profits ^c				
		Across the Incubator Tank	Across the Flash Heater	Across the Clarifier Tank	Across the Evaporator Station	Total
Season Average	Cold		\$176,920	\$222,178	\$209,834	\$608,932
	Interm		\$94,640	\$213,949	\$176,920	\$485,509
	Hot	\$18,515	n/a	\$181,034	\$126,724	\$326,273

^c U.S. dollar losses were calculated taking into account the pounds of raw sugar produced by the factory in 2000, the average cane sugar recovery rate, the average sucrose content of the raw sugar, and the current average price of raw sugar (19c per lb).

No sucrose losses were detected by the preheating of acidic MJ in intermediate and hot liming. Even though juice retention time was 8 min in the cold and intermediate incubation tank, no sucrose losses were detected (Table 3a), but there was a slight amount of sucrose lost in the hot lime incubation tanks where the retention time was 4 min longer at 12 min. This suggests that 12 min retention time may be too long and that 8 min should be adequate. A marked amount of sucrose (range 0.25-0.64%) was lost across the flash heater in cold liming (Table 3a) which caused a season average loss in revenue of US \$176,920 (Table 3b). In contrast, because of the design of the hot lime clarification process (Fig. 1a), losses across the flash heater were not applicable, and sucrose losses across the flash heater in intermediate liming were only detected in the first and final sampling periods (Table 3a). These differences may have been because of the slower settling in cold liming (see settling section), and the extra sucrose losses across the cold lime flash heater are an obvious disadvantage.

The effect of increasing the FES target pH from 6.0 to 6.3 for both intermediate and hot liming in sampling period 3, markedly decreased sucrose losses and increased economic profits, particularly across the evaporator station (Table 3). Furthermore, this positive effect was stronger for hot rather than intermediate liming. One of the reasons raw sugar factory staff are reluctant to increase either the target pH of the clarified juice or final evaporator syrup because they believe the extra lime required would increase evaporator scaling, particularly in the later evaporator bodies. However, the majority of unwanted scaling, especially in the U.S., is usually due to the precipitation of insoluble silicates, which are mostly associated with cane, soil, and trash entering the factory. Moreover, the markedly less lime required in hot liming would offset the additional lime required to increase the pH and improve sucrose inversion losses across the evaporators.

Overall across the season (Table 3b), the use of hot liming approximately saved the factory nearly half of the profits they would have lost if they operated cold liming. Total season losses in profits (Table 3b) for intermediate liming (US \$485,509), were better than in cold liming (US \$608,932) but worse than in hot liming (US \$326,273).

Turbidity Removal and Settling Performance

On season average, there was approximately 2.3% (significant at $P < 0.05$) more turbidity removal (MJ to CJ) in both hot and intermediate liming than in cold liming (Table 4). This was slightly lower than the 4.6% difference observed by Eggleston *et al.* (2002) between intermediate and cold liming, across the 1999 grinding season. The lower removal in this study may be because of different cane quality, as the factory processed considerably more

green than burnt billeted cane than in the previous year, which increases the load of impurities. Turbidity values for clarified juices in both intermediate and hot liming were significantly ($P < 0.05$) lower than in cold liming (Table 4) and this was further reflected in the final evaporator syrup and raw sugar turbidity values (Table 4). Turbidity control was also markedly better in the hot liming FES and raw sugars.

TABLE 4.
SEASON DIFFERENCES IN TURBIDITY VALUES AND REMOVAL^a

Sample	Turbidity at 420 nm (ICU) ^b		
	Cold	Intermediate	Hot
MJ	57153 \pm 10959a ^c	60283 \pm 8014a	59437 \pm 5504a
CJ	3165 \pm 454a	1966 \pm 354b	2100 \pm 333b
FES	6079 \pm 911a	5022 \pm 762b	4868 \pm 358b
Raw Sugar	755 \pm 252	693 \pm 311	445 \pm 24
Av. % Turbidity Removal: MJ to CJ	94.5	96.7	96.5

^a N = 18 except for hot liming where N = 12

^b Season average data presented with standard deviations

^c Lower case letters represent statistical differences ($P < 0.05$) between the three clarification processes for season averages

The similarity in turbidity removal for hot and intermediate liming was expected because both processes had 50% of the MJ preheated before incubation. It is well known that the preheating of cane juice increases floc size through coagulation (Eggleston *et al.* 2002), and larger flocs settle faster. However, differences were apparent in the settling performance of the flocculated limed juices from the three clarification processes (Table 5). Across the season, visually the flocs were generally large in intermediate liming, compared to moderately large in hot liming and medium to small, fine flocs in cold liming (Table 5). The visually larger flocs in intermediate liming caused settling to be faster, as indicated by the higher initial settling rates and lower break point times (Table 5). Reasons for the larger flocs in intermediate compared to hot liming are not clear; the only differences between the two processes were the higher temperature and lower retention time of lime addition in the hot liming. The addition of lime at higher temperatures and for a shorter time in hot liming may have ruptured some of the flocs formed previously on preheating the MJ. Another explanation could be that the formation of calcium

phosphate precipitate was lower in hot liming because of the shorter liming time. Overall, results strongly suggest that the preheating of juice is a large contributor to settling performance and probably contributes more to what was previously considered (Simpson 1996).

TABLE 5.
SETTLING CHARACTERISTICS OF THE THREE CLARIFICATION PROCESSES^a

Clarification Method	Sampling Period	ISR ^b (ml/min)	Break Point ^c (secs)	MV _{inf} ^d (ml)	Visual Floc characteristics
Cold	1	94.3	41.3	13.9	Medium to large flocs
	2	108.3	41.2	12.1	Small, fine flocs
	3	95.7	43.5	14.5	Small, fine flocs
Intermediate	1	115	39	10.1	Mostly large flocs
	2	140	33.3	9.6	Large flocs
	3	180 ^e	30.2	10.7	Large to v. large flocs
Hot	1	--	--	--	--
	2	103.8	43.3	12.2	Moderately large flocs
	3	91.4 ^f	53.5	12.0	Medium to large flocs

^a Averages \pm standard errors; N \leq 6

^b ISR = initial settling rate. This was calculated from the initial slope of mud volume (mL) versus time (min), and reflects the rate of settling in the clarification tank.

^c Break Point was the time in seconds it took for the flocculated, flash heated limed juice to settle to half its original volume

^d The final equilibrium mud volume at infinity MV_{inf} or final height of the mud after infinite time, was obtained from the intercept of a plot of % mud volume versus 1/t, where t is time in minutes

^e N \leq 4

^f N \leq 2 due to difficulties of measuring settling in some samples

Effect on Polysaccharide Concentrations

The two major polysaccharides which can profoundly impact cane processing are dextran and starch. Dextran is formed from cane contamination with *Leuconostoc* bacteria. Starch is present in the cane as a storage source and is less abundant in mature than immature cane.

Dextran removal and formation (Table 6), in general, followed previous observations by Eggleston *et al.* (2002). One of the advantages of preheating MJ before liming, as in intermediate and hot lime processes, is the removal of polysaccharides including dextran, which also cause the levels to be lower in the incubated juice. However, a marked drop of dextran was also noted in the incubation tank when the cold liming process was operated (Table 6), which suggests that some precipitation occurred with color removal. As expected, dextran was markedly formed in the lime tanks for the cold liming process (Table 6). Dextran concentrations across the intermediate lime process were not as good as in the previous season, but hot liming levels were much better although it must be pointed out that the incoming MJ levels were better too.

TABLE 6.
SEASON AVERAGE DEXTRAN (ASI-II METHOD) DATA^a

Sample	Dextran (ppm/Brix) ^b		
	Cold	Intermediate	Hot
MJ	790 ± 480a ^c	804 ± 513a	551 ± 136a
HJ	N/A	459 ± 277a	400 ± 185a
Incub J	529 ± 317	717 ± 539	582 ± 187
LJ/HLJ	1001 ± 550a	680 ± 451a	N/A
FHLJ	362 ± 112a	697 ± 716a	361 ± 118a
CJ	357 ± 183a	492 ± 483a	295 ± 140a
FES	467 ± 172a	582 ± 428a	274 ± 65a
Raw Sugar	432 ± 315	356 ± 88	158 ± 74

^a N=18 except for hot liming samples where N=12

^b Season average data presented with standard deviations

^c Lower case letters represent statistical differences ($P < 0.05$) between the three clarification processes for season averages

As expected (Eggleston *et al.* 2002), starch decreased across the season for all samples and clarification systems, because of the increased maturity of cane being processed. MJ starch varied little for the three clarification processes (Fig. 5). In both intermediate and hot liming, starch was removed in the HJ most likely because of precipitation. Starch was also degraded in the incubator tank for both intermediate and hot liming (Fig. 5) which is most likely because the addition of recycled filtrate from the clarifier reduced the MJ acidity, enabling

the natural juice diastase to degrade starch (Eggleston *et al.* 2002). Although, starch increased for all three processes in the clarified juice it is not possible for starch to increase during clarification, it can only be chemically or physically removed during the process. Currently, therefore, these results, are not fully understandable and further investigation is warranted. The increases may be a symptom of the starch method used or could somehow be caused by the prolonged high temperatures in the clarifier. Starch decreased in the FES because of the factory application of commercial α -amylase.

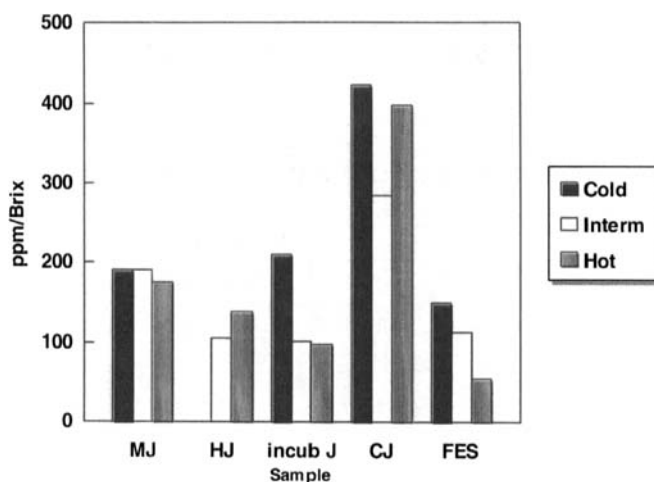


FIG. 5. VARIATIONS IN SEASON AVERAGE STARCH CONCENTRATIONS AMONG THE THREE CLARIFICATION PROCESSES

MJ=mixed juice; HJ=heated juice; Incub J=incubated juice; CJ=clarified juice;
FES=final evaporator syrup

CONCLUSIONS

For most clarification parameters investigated, both hot and intermediate liming performed much better than cold liming, and hot liming generally offered some extra advantages over intermediate liming. Furthermore, after operating hot liming across the 2000 grinding season, the factory observed 12-15% more heating capacity in the limed juice heat exchangers and a 90% reduction in the quantity of chemicals needed to clean the heat exchangers. Most of the advantages offered by hot and intermediate liming were because of (1) preheating the juice before incubation and liming which markedly improves impurity removal, and (2) in the case of hot liming, the much lower consump-

tion of lime. To gain these advantages it is not necessary for a sugarcane factory to have an incubator tank. However, an incubation tank does offer some benefits such as increased starch removal and stabilization of factory juice flow rate. It is also important to point out that, in hot liming, the lime does not have to be added after the flash heater, but can be added just before the heater and this could increase mixing. In South Africa, many factories add lime upstream of the flash tank in an in-line static mixer (Meadows 1996).

ACKNOWLEDGMENTS

The American Sugar Cane League is gratefully acknowledged for funding this study. Thanks to E. St. Cyr and J. Sanchez for excellent technical assistance, and to Analytical Laboratories, Inc., LA and Galbraith Laboratories, TN, for undertaking the calcium and silicate determinations, respectively. The authors are grateful to G. Myers of Louisiana State University for help in undertaking the statistical analyses. Thanks to P. Rein of the Audubon Sugar Institute for useful discussions.

REFERENCES

- ARMAS DE, R., MARTINEZ, M., VICENTE, C. and LEGAZ, M.E. 1999. Free and conjugated polyamines and phenols in raw and alkaline clarified sugarcane juices. *J. Agric. Food Chem.* **47**, 3086–3092.
- BENNETT, M.C. and RAGNAUTH, J.M. 1960. The effects of calcium and phosphate in cane juice clarification. *Intern. Sugar J.* **62**, 13–16.
- BOYES, P.N. 1960. Starch in the manufacture of raw sugar. *Proc. Ann. Congr. S. Afr. Sugar Technol. Assoc.* **34**, 91.
- BUCHELI, C.S. and ROBINSON, S.P. 1994. Contribution of enzymic browning to color in sugarcane juices. *J. Agric. Food Chem.* **42**, 257–261.
- CHEN, J.C.P. 1993. Purification of the juice. Part (A) clarification reaction and control. In *Cane Sugar Handbook* 12th Ed., (J.C.P. Chen and C.C. Chou, eds.) pp. 103, John Wiley & Sons, New York.
- CLARKE, M.A., ROBERTS, E.J. and THANH, B.T. 1986. Recent studies on dextrans and polysaccharides in refinery processes. *Proc. Sugar Proc. Res. Conf., Savannah, GA*, 74–92.
- EGGLESTON, G. 2000a. Hot and cold lime clarification in raw sugar manufacture. I: Juice quality differences. *Intern. Sugar J.* **102**, 406–416.
- EGGLESTON, G. 2000b. Hot and cold lime clarification in raw sugar manufacture. II: Lime addition and settling behaviour. *Intern. Sugar J.* **102**, 453–457.

- EGGLESTON, G., CLARKE, M.A. and PEPPERMAN, A.B. 1999. Mixed juice clarification of fresh and deteriorated sugarcane. *Intern. Sugar J.* 101, 296-300, 341-344.
- EGGLESTON, G., MONGE, A. and PEPPERMAN, A. 2002. Pre-heating and incubation of cane juice prior to liming: A comparison of intermediate and cold liming. *J. Agric. Fd. Chem.* 50, 484-490.
- GODSHALL, M.A., CLARKE, M.A. and DOOLEY, C.D. 1991. Starch: process problems and analytical developments. *Proc. 1990 Sugar Proc. Res. Conf.*, pp. 244-264, San Francisco, CA.
- LILLEHOJ, E.B., CLARKE, M.A. and TSANG, W.S.C. 1984. *Leuconostoc* SPP in sugarcane processing samples. *Proc. Sugar Processing Research Conf.*, pp. 141-151, New Orleans, LA.
- MEADOWS, D.M.C. 1996. Raw juice flow control, screening, heating and liming. *Proc. Ann. Congr. S. Afr. Sugar Technol. Assoc.* 70, 272-276.
- MULLER, C. 1921. Cause and remedy of the difficult defecation of cane juice. *Intern. Sugar J.* 23, 679-681.
- SARKAR, D. and DAY, D.F. 1986. Dextran analysis: A modified method. *J. Am. Soc. Sugar Cane Technol.* 6, 102-107.
- SCHAFFLER, K.J., MUZZELL, D.J. and SCHORN, P.M. 1985. An evaluation of sucrose inversion and monosaccharide degradation across evaporation at Darnall mill. *Proc. Ann. Congr. S. Afr. Sugar Technol. Assoc.* 59, 73-78.
- SIMPSON, R. 1996. The chemistry of clarification. *Proc. Ann. Congr. S. Afr. Sugar Technol. Assoc.* 70, 267-271.